

Effect of a Novel Family of *N,N'*-Diphenyl Bisamides on the Formation of β Crystalline Form in Isotactic Polypropylene

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In this paper, a family of eight *N,N'*-diphenyl bisamide compounds were synthesized and characterized by fourier transform infrared (FT-IR) and ^1H nuclear magnetic resonance (NMR) technologies. The influence of different compounds on the formation of β crystalline form in iPP had been investigated by means of wide angle X-ray diffraction (WAXD), differential scanning calorimetry (DSC) and polarized light microscopy (PLM). *N,N'*-diphenyl succinamide (DPS), *N,N'*-diphenyl glutaramide (DPG) and *N,N'*-diphenyl adipamide (DPA) are β -nucleating agents for iPP among these compounds. The results also indicate that DPS and DPA have higher ability to induce the β -form than DPG. The analysis of the cell parameters of three efficient β -nucleating agents shows that they all have approximate periodic length of *b*-axis of 0.65 nm. And it is also suggested that two-dimensional lattice matching relationships exist between the crystal structures of the β -nucleating agents and that of β -iPP.

KEY WORDS: Polypropylene / Polymorphism / Epitaxy / Nucleating Agent / β Crystalline Form /

Isotactic polypropylene (iPP) is an excellent thermoplastic for its desirable thermal stability and mechanical property, which makes iPP become an extremely active research object. The physical properties of materials based on the iPP strongly depend upon its morphology, macromolecular structure, loading condition, etc.^{1–3} As widely reported, iPP can form four crystalline structures: α , β , γ and smectic forms under different conditions.^{4–6} The common α -phase can be promoted with addition of nucleating agents.⁷ Meanwhile, a predominant β phase within iPP can be obtained by shearing field, temperature gradient and specific β nucleating agents.^{8–13} Pure or nearly pure β phase had been obtained by Varga *et al.*,^{14,15} though β phase is meta-stable^{16,17} and it may convert into α -phase while the specimen is applied with heating treatment or mechanical force.^{18,19}

As widely acknowledged, the trigonal β -phase shows 3₁ helical hand which is the same as α - and γ -phases.^{20,21} The assembly of macromolecular chains of iPP towards the given crystalline structure is affected by the crystallographic interaction between iPP and the nucleating agent. For β -phase iPP, Lotz and co-workers^{22,23} discussed the crystalline structural relationship between the nucleating agent and iPP, and proposed “dimensional lattice matching theory” to explain this phenomenon. They found that nucleating agent with about 0.65 nm periodicity and an orthogonal geometry of the contact face is prone to induce the formation of β -phase iPP.²⁴ On the other hand, according to the observation of atomic force microscopy, β -lamellae epitaxially crystallized on the lateral planes of the β -nucleating agent.^{25,26}

Compared with the plentiful α -nucleating agents, the efficient β -nucleating agents for iPP are rare, quinacridone pigment, pimelic acid/calcium stearate mixture, calcium salts of suberic or pimelic acid, *N,N'*-dicyclohexyl-terephthalamide,

N,N'-dicyclohexyl-2, 6-naphthalene-dicarboxamide are the most frequently used,⁹ which limits the study on the nucleation mechanism in depth. There is no systemic investigation on the relationship between *N,N'*-diphenyl bisamide compounds and the polymorphism of iPP. The objective of this paper is to study the influence of different bisamide compounds on the formation of β -form iPP, and to further discuss the relationship between the crystal structures of the amide nucleating agents and that of β -iPP.

EXPERIMENTAL

Materials

IPP powder (MFR = 1 g/10 min at 230 °C and 21.2 N load) was obtained from Jingling Plastics and Rubber Chemical Co, Ltd. Industrial antioxidant (B215) was commercially available. Aliphatic dicarboxylic acid and phenylamine (analytical grade reagents) were used to synthesize a family of bisamide compounds, and they were characterized by fourier transform infrared spectrometer (VECTORTM 22, Bruker, Germer) and ^1H NMR spectrometer (AVANCE 400D+ HR/MAS, Bruker, Switzerland) after purification. The FT-IR and ^1H NMR data of the eight amide compounds are listed below:

N,N'-Diphenylmalonamide (DPM). FT-IR (KBr): 3271.66 cm^{-1} (NH), 3150.01 cm^{-1} (ϕ -H), 168.18 cm^{-1} and 1647.55 cm^{-1} (C=O), 1444.02 cm^{-1} and 1414.13 cm^{-1} (ϕ -N), 1597.54 cm^{-1} and 1537.81 cm^{-1} (CH). ^1H NMR (DMSO-*d*₆, 400 MHz): δ = 3.44 (t, 2H), δ = 7.08 (m, 2H), δ = 7.32 (m, 4H), δ = 7.63 (m, 4H), δ = 10.24 (m, 2H).

N,N'-Diphenylsuccinamide (DPS). FT-IR (KBr): 3307.99 cm^{-1} (NH), 3056.01 cm^{-1} (ϕ -H), 1705.36 cm^{-1} and 1658.11 cm^{-1} (C=O), 1340.78 cm^{-1} and 1288.27 cm^{-1} (ϕ -N), 1527.92 cm^{-1} and 1499.79 cm^{-1} (CH). ^1H NMR (DMSO-*d*₆, 400 MHz): δ =

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2.65 (s, 2H), $\delta = 7.02$ (m, 2H), $\delta = 7.26$ (m, 4H), $\delta = 7.40$ (t, 4H), $\delta = 9.97$ (s, 4H).

N,N'-Diphenylglutaramide (DPG). FT-IR (KBr): 3310.94 cm^{-1} (NH), 3080.78 cm^{-1} (ϕ -H), 1745.15 cm^{-1} and 1673.18 cm^{-1} (C=O), 1319.15 cm^{-1} and 1270.75 cm^{-1} (ϕ -N), 1549.92 cm^{-1} and 1500.32 cm^{-1} (CH). ^1H NMR (DMSO- d_6 , 400 MHz): $\delta = 1.90$ (m, 2H), $\delta = 2.37$ (t, 4H), $\delta = 7.01$ (t, 2H), $\delta = 7.28$ (t, 4H), $\delta = 7.59$ (d, 4H), $\delta = 9.89$ (s, 2H).

N,N'-Diphenyladipamide (DPA). FT-IR (KBr): 3245.95 cm^{-1} (NH), 3076.01 cm^{-1} (ϕ -H), 1658.18 cm^{-1} (C=O), 1553.45 cm^{-1} and 1488.16 cm^{-1} (CH) 1443.95 cm^{-1} and 1385.31 cm^{-1} (ϕ -N). ^1H NMR (DMSO- d_6 , 400 MHz): $\delta = 1.56$ (m, 4H), $\delta = 2.34$ (m, 4H), $\delta = 7.18$ (t, 2H), $\delta = 7.28$ (t, 4H), $\delta = 7.59$ (d, 4H), $\delta = 9.87$ (s, 2H).

N,N'-Diphenylheptanediamide (DPH). FT-IR (KBr): 3297.67 cm^{-1} (-NH), 2945.15 cm^{-1} (ϕ -H), 1666.62 cm^{-1} and 1597.93 cm^{-1} (C=O), 1538.62 cm^{-1} and 1498.64 cm^{-1} (CH), 1443.24 cm^{-1} and 1414.01 cm^{-1} (ϕ -N). ^1H NMR (DMSO- d_6 , 400 MHz): $\delta = 1.34$ (m, 2H), $\delta = 1.62$ (m, 4H), $\delta = 2.30$ (t, 4H), $\delta = 7.01$ (m, 2H), $\delta = 7.25$ (t, 4H), $\delta = 7.47$ (d, 4H), $\delta = 9.93$ (s, 2H).

N,N'-Diphenyloctanediamide (DPO). FT-IR (KBr): 3300.12 cm^{-1} (NH), 2942.26 cm^{-1} (ϕ -H), 1657.21 cm^{-1} and 1598.06 cm^{-1} (C=O), 1535.91 cm^{-1} and 1498.62 cm^{-1} (CH), 1442.73 cm^{-1} and 1420.64 cm^{-1} (ϕ -N). ^1H NMR (DMSO- d_6 , 400 MHz): $\delta = 1.33$ (t, 4H), $\delta = 1.59$ (t, 4H), $\delta = 2.28$ (t, 4H), $\delta = 7.00$ (t, 2H), $\delta = 7.27$ (t, 4H), $\delta = 7.58$ (d, 4H), $\delta = 9.91$ (s, 2H).

N,N'-Diphenylnonanediamide (DPN). FT-IR (KBr): 3258.01 cm^{-1} (NH), 2937.87 cm^{-1} (ϕ -H), 1668.36 cm^{-1} and 1598.52 cm^{-1} (C=O), 1552.38 cm^{-1} and 1501.05 cm^{-1} (CH), 1442.95 cm^{-1} and 1414.72 cm^{-1} (ϕ -N). ^1H NMR (DMSO- d_6 , 400 MHz): $\delta = 1.30$ (s, 6H), $\delta = 1.58$ (t, 4H), $\delta = 2.29$ (t, 4H), $\delta = 7.00$ (t, 2H), $\delta = 7.26$ (m, 4H), $\delta = 7.57$ (d, 4H), $\delta = 9.83$ (s, 2H).

N,N'-Diphenyldecanediamide (DPD). FT-IR (KBr): 3297.69 cm^{-1} (NH), 2929.81 cm^{-1} (ϕ -H), 1652.22 cm^{-1} and 1597.55 cm^{-1} (C=O), 1525.75 cm^{-1} and 1498.41 cm^{-1} (CH), 1443.10 cm^{-1} and 1420.04 cm^{-1} (ϕ -N). ^1H NMR (DMSO- d_6 , 400 MHz): $\delta = 1.29$ (s, 8H), $\delta = 1.58$ (t, 4H), $\delta = 2.28$ (t, 4H), $\delta = 7.00$ (t, 2H), $\delta = 7.27$ (t, 4H), $\delta = 7.57$ (d, 4H), $\delta = 9.83$ (s, 2H).

Preparation of the Nucleated IPP Samples

IPP powder (200 g) was mixed with B215 (0.2 g) and variable amounts of the nucleating agent in a mixer at a speed of 25000 rpm for 2 min. The mixtures were compounded in a single-screw extruder (LSJ20, D = 20 mm, L/D = 25/1, Shanghai KeChuang Rubber Plastic's Machinery Set Co. Ltd). The rate of the screw was 60 rpm. The temperature of the extruder from the hopper to the die was set at 210 °C, 220 °C, 220 °C and 210 °C, respectively. The melt was cooled and pelletized. IPP doped without the nucleating agent was also prepared in a similar way to produce a blank control sample.

A pellet of the blank or the nucleated iPP sample was placed between two glass slides on a hot stage kept at 220 ± 2 °C for

10 min to allow the sample to melt completely and remove thermal memory. Pressure was then applied on the top slide to form a film, which was then immersed in a glycerin bath set at 120 ± 1 °C, isothermally crystallized for 30 min, and then quenched in iced water. The thickness of samples is *ca.* 0.5 mm for wide angle X-ray diffraction (WAXD) and differential scanning calorimetry (DSC) characterization and *ca.* 10~20 μm for polarized light microscopy (PLM) observation, respectively.

Wide Angle X-ray Diffraction (WAXD) Characterization

WAXD diffraction patterns of the blank or the nucleated iPP samples were recorded on an X-ray diffractometer (ARL X'TRA, Thermo Electron Corporation, USA) using Cu K α radiation ($\lambda = 0.154$ nm). The diffractometer was operated in reflection mode at 45 kV and 35 mA. Radial scans of intensity vs. diffraction angle (2θ) were recorded in the range of 5 to 40°. The scanning rate was 10°/min.

The characteristic crystal planes for the monoclinic α -form iPP are: (110), (040), (130) and (041) planes, which correspond to the diffraction angles $2\theta = 14.2^\circ$, 17.0° , 18.6° and 22.0° , respectively. And the characteristic crystal planes (300) and (301) for β -form are characterized at the diffraction angles $2\theta = 16.2^\circ$ and 21.2° . The relative proportion of β -phase in iPP sample was measured by the empirical ratio K_{WAXD} :²⁷

$$K_{\text{WAXD}} = \frac{H(300)}{H(300) + H(110) + H(040) + H(130)} \quad (1)$$

where $H(300)$, $H(110)$, $H(040)$ and $H(130)$ are the heights of the β -form peak (300) and three strong α -form peaks (110), (040) and (130), respectively. All the heights of the peaks were measured above the amorphous background. The total degree of crystallinity (X_{WAXD}) of the sample was calculated by the function as below:

$$X_{\text{WAXD}} = \frac{I_C}{I} \times 100 \quad (2)$$

where I_C was the integral intensities diffracted by the crystalline parts, and I was the total integral intensities.²⁸

Differential Scanning Calorimetry (DSC) Measurement

The melting behaviors of the blank iPP and the nucleated iPP samples were measured in a DSC instrument (CDR-34P, Shanghai Precision Scientific Instrument Co. Ltd) under a dry nitrogen atmosphere. About 5 mg of each sample was sealed in an aluminium pan and heated from room temperature to 220 °C at a rate of 10 °C/min. The heat flow and the temperature were recorded. The peak melting temperatures of α form ($T_{m,\alpha}$) and β -form ($T_{m,\beta}$) were obtained from the melting curves. The enthalpy of the α -form (H_α) and β -form (H_β) were obtained from the separation of the areas of the α - and β -form melting peaks. The content of β -form was calculated by the function as below:

$$K_{\text{DSC}} = H_{m,\beta} / (H_{m,\beta} + H_{m,\alpha}) \quad (3)$$

Polarized Light Microscopy (PLM) Observation

The spherulitic structure of the blank and the nucleated iPP samples were observed by a polarized light microscope (LW-200-4JS, Shanghai LW Scientific Co. Ltd) equipped with cross polars and a CCD camera. Images were captured and stored in a computer. The signs of birefringence of the spherulites were determined by means of a primary red filter (λ -plate) located diagonally between cross polars. In this way the first and third quarters of the sight are yellow and the second and fourth are blue when the spherulites are negative, while a reversed arrangement of the quarters are observed for positive spherulites.

RESULTS AND DISCUSSION

WAXD Measurements

The suitable temperature range for the formation of β -form iPP (the growth rate of β -form is faster than that of α -form, *i.e.*, $G_\beta > G_\alpha$) is between 105 °C and 140 °C.^{9,29} Nakamura *et al.*³⁰ found the two crossover temperatures are 90 °C and 133 °C for low tacticity iPP. And in a previous paper, we found that the optimum temperature for the formation of β -form iPP was about 120 °C.³¹ Thus we chose 120 °C as the crystallization temperature for the samples in this paper.

WAXD patterns of various bisamides/iPP systems are shown in Figure 1. Compared with the blank iPP sample, the samples doped with DPS, DPG and DPA show the strong diffraction peaks at $2\theta = 16.2^\circ$ and 21.2° corresponding to β -form iPP, suggesting predominant β -forms exist in the samples. On the contrary, the samples doped with DPH, DPO and DPD show the existence of few or none β -forms, and the samples doped with DPM and DPN show the existence of a small amounts of β -forms. It is shown that DPS, DPG and DPA have the ability to induce the β -form within iPP samples under the present condition. According to eq (1), the relative proportion of β -form (K_{WAXD}) in the iPP samples are listed in Table I. For DPS/iPP, DPG/iPP and DPA/iPP systems, the K_{WAXD} values are 94.7%, 71.3%, 89.9%, respectively. Moreover, DPS/iPP and DPA/iPP systems presented greater proportion of β -form

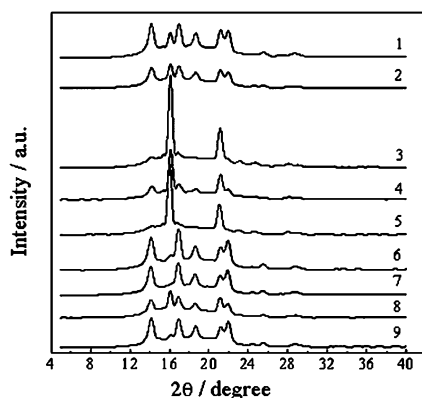


Figure 1. WAXD patterns of the blank and the nucleated iPP with addition of 0.1 wt% different amide compounds. 1: blank iPP; 2: DPM; 3: DPS; 4: DPG; 5: DPA; 6: DPH; 7: DPO; 8: DPN; 9: DPD.

Table I. The K_{WAXD} and X_C of the blank iPP and the nucleated iPP with addition of 0.1 wt% different amide compounds

	iPP	DPM	DPS	DPG	DPA	DPH	DPO	DPN	DPD
K_{WAXD} (%)	20.2	33.7	94.7	71.3	89.9	5.9	0	42.0	9.1
X_C (%)	48.7	47.1	61.3	50.9	56.5	50.4	48.8	47.6	49.9

than DPG/iPP system, while the three systems contain the same concentration level. In other words, DPS and DPA have a higher ability to induce the formation of β -form than DPG.

Meanwhile, the total degrees of crystallinity (X_C) of all samples are also listed in Table I. It is shown that different amide compounds bring different influence on the crystallinity. Three effective β -nucleating agents not only change the crystal structure, but also improve the crystallinity of iPP. It is indicated that DPS, DPG and DPA can provide nucleating centers and accelerate the macromolecular chains to form regular structure.

DSC Measurement

To further study the effect of different amide compounds on the proportion of β -form within iPP samples, DSC measurements were also utilized (Figure 2). The melting curves of samples containing rich β -form exhibit two melting peaks. The first one corresponds to β -form, while the second one corresponds to α -form. The melting enthalpy of α -form (H_α) and β -form (H_β) for different compound/iPP systems are listed in Table II. The results of K_{DSC} also show that DPS/iPP, DPG/iPP and DPA/iPP systems present great proportion of β -form, which are accordant with the results of WAXD. However,

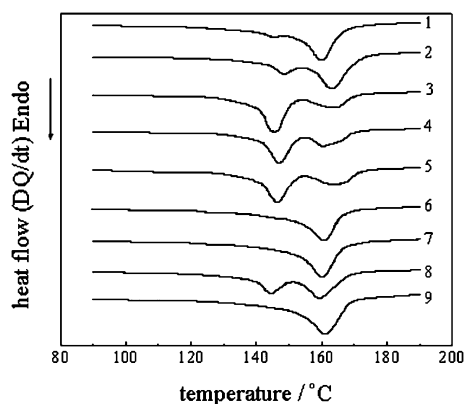


Figure 2. DSC melting curves of the blank and the nucleated iPP with addition of 0.1 wt% different amide compounds. 1: blank iPP; 2: DPM; 3: DPS; 4: DPG; 5: DPA; 6: DPH; 7: DPO; 8: DPN; 9: DPD.

Table II. The melting points, enthalpies and K_{DSC} values of the blank iPP and the nucleated iPP with addition of 0.1 wt% different amide compounds

	iPP	DPM	DPS	DPG	DPA	DPH	DPO	DPN	DPD
$T_{m,\alpha}$ (°C)	161.5	164.9	165.9	163.1	165.6	162.9	161.9	161.7	162.7
H_α (J/g)	108.3	124.9	61.7	65.2	65.7	128.5	141.7	111.1	154.0
$T_{m,\beta}$ (°C)	146.9	148.6	147.6	148.7	148.2			146.1	
H_β (J/g)	21.2	59.2	117.9	94.2	100.7			74.6	
K_{DSC} (%)	15.7	32.7	65.6	59.1	60.5			40.2	

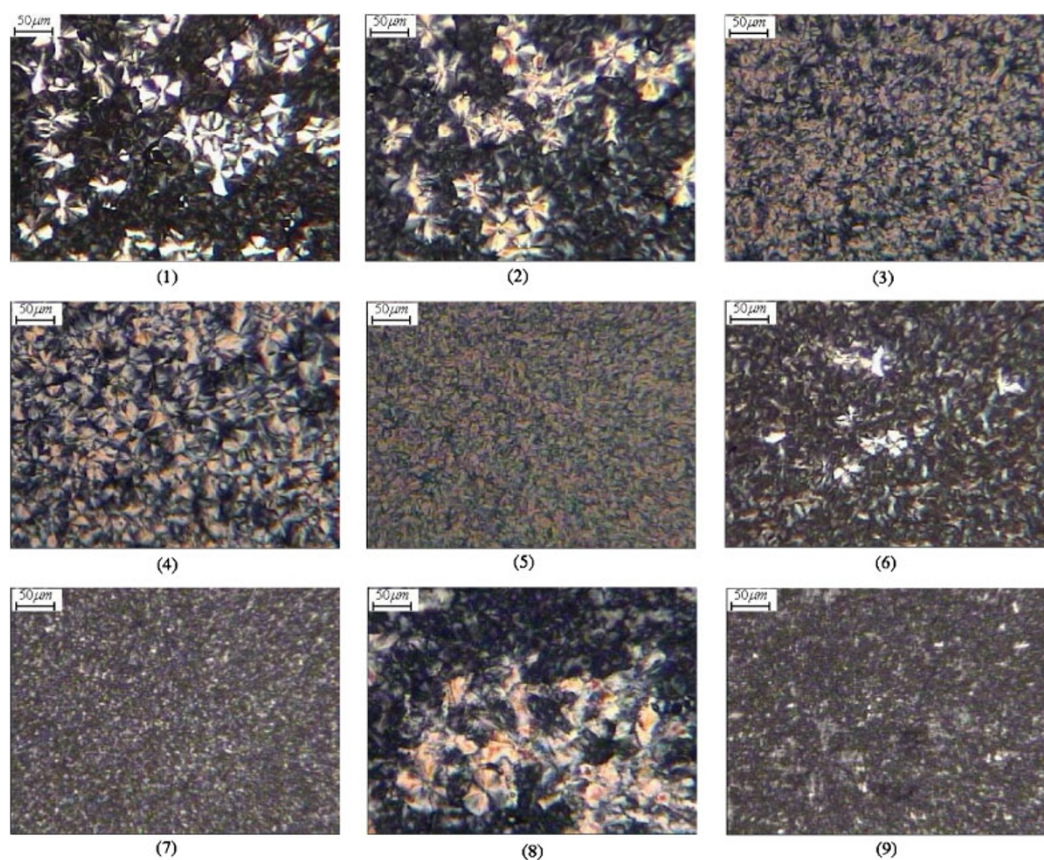


Figure 3. The PLM images of the blank and the nucleated iPP with addition of 0.1 wt % different amide compounds. (1): blank iPP; (2): DPM; (3): DPS; (4): DPG; (5): DPA; (6): DPH; (7): DPO; (8): DPN; (9): DPD.

while β -form goes through the heating process, it will transform into α -form easily. And that is associated with the phenomena that the second melting peaks of curves 3, 4 and 5 show wide than the others, and curves 6 and 9 do not present the melting peaks corresponding to β -form iPP. This transformation process can be described as: the β -form melts firstly; the new α -form recrystallizes from the original β -form; α -form melts finally.^{14,32–35}

PLM Observation

The β -form iPP exhibits bright or color spherulitic morphology, and is very easy to be distinguished from the black and white α -form under polarized field. β spherulites show strong negative sign of birefringence when a primary red filter (λ -plate) is inserted diagonally between the crossed polarizers, which are in accord with the result of the literature.⁹ From the images of (3), (4) and (5) in Figure 3, two phenomena can be seen clearly: more numbers of colored β -spherulites and smaller β -spherulitic sizes. The addition of DPS, DPG and DPA can support more nucleating centers, which give rise to more numbers of small spherulites; then the impingement between the spherulites increased; consequently the development of spherulites is limited. In the image (7) of Figure 3, no visible β -form but fine α spots can be seen. That indicates DPO may suppress the formation of β -form iPP, and this observation is accordant with the results of WAXD and DSC.

β Nucleation Mechanism

Beck³⁶ proposed a good nucleating agent should possess at least the five following characteristics. (a) It should be capable of reducing the interfacial surface free energies involved, *i.e.*, it should be wet by polypropylene or adsorb polymer on its surface at and below the polymer melting point. (b) It should be insoluble in polypropylene at and below the polymer melting point. (c) It should melt above the polymer melting point and preferably without decomposition. (d) It should be nonvolatile, stable, and unreactive towards its environment, *i.e.*, polymer, oxygen, moisture, and miscellaneous polymer additives. (e) Also, if possible, it should possess a crystalline structure similar to that of polypropylene. A model nucleating agent for PP consisting essentially of two parts: an organic group (solubilizing or “wetting” portion, such as hydrocarbon) and a polar group (insolubilizing portion, for example, a carboxylic acid moiety). Binsbergen^{37,38} proposed nucleating agents for polyolefins are crystalline. Most of the agents are insoluble in the polymer melt. And the agents consist of both hydrocarbon groups (resembling good solvents for the polymers) and either polar groups or a condensed aromatic structure (rendering the agents insoluble in the polymer melt). For eight amide compounds discussed in this paper, they all are highly ordered crystalline compounds, and they have phenyl and amide group (rendering the agents insoluble in the polymer melt) and aliphatic chain (resembling good solvents for PP melt) in their

molecular structures. Thus they can be wet by polypropylene melt, but are not dissolve in the melt. During cooling from the melt, these compounds reduce the activation free energy for nucleation and provide a large number of sites for the initiation of crystallization, producing a more uniform crystalline structure and a higher degree of crystallinity.

From Table I, it is found that K_{WAXD} and X_C values of the samples nucleated by DPS, DPG and DPA are greater than those of the others. Why only three of these bisamides are good β nucleating agents for iPP? Obviously, it relates to their molecular and crystalline structures. Beck³⁶ also found that the dependence of nucleating ability on the chain length of aliphatic mono- and dicarboxylates and alicyclic monocarboxylates. He thought it possibly reflects an ability of the compound through favorable geometry to form a crystal lattice of optimum surface energy and/or optimum dimensions for nucleation and epitaxial growth of polypropylene.

Epitaxy was ruled out as a mechanism for the nucleating effect by Binsbergen.³⁸ But Galeski³⁹ indicated the extensive data on the nucleating agent of crystallization of PP do not completely satisfactorily match the theory of heterogeneous nucleation which is based on the surface free-energy consideration. The important feature of a good nucleating agent appears to be the existence of a crystal lattice matching for epitaxial growth of PP. At present, the epitaxial crystallization mechanism has been widely used to explain the way of inducing a polymer crystal growing on the surface of a nucleating agent. And this process may be influenced by the molecular structure or crystallographic morphology of the nucleating agent.

Since different nucleating agent may provide different contacting surface for the growth process of β -form. Thereby, the crystal structure of different compound should be considered. The amide group is an excellent proton donor, and the carbonyl group is a proton acceptor. Thus, the connective pattern between molecules is *via* N-H...O hydrogen bond and this strong connection make the torsion angle of N-H...O tends to near-linearity.⁴⁰ Another important factor affecting the crystalline structure is steric hindrance of carbon chain which is dependent on the length of carbon chain of this family. The cell parameters of the eight compounds calculated from their powder WAXD data are given in Table III. It is found that DPS, DPG and DPA have a similar periodic length of b -axis of *ca.* 0.65 nm, which is the periodic length of c -axis of β -iPP. But the other five compounds do not show the close values. Stocker *et al.*²⁴ also found this interesting phenomenon, and pointed out that the substrate of 0.65 nm periodicity was parallel to and matched the iPP chain-axis repeat distance.

In terms of the misfit factor (f_m) function, shown as below:

$$f_m = 100 \times \frac{PB - PA}{PA} \quad (4)$$

in which PA and PB are the appropriate period length of the substrate and polymer respectively, f_m has an upper limit reported as 15% empirically.⁴¹ The misfit factors between the lengths of b -axis of DPS, DPG, DPA and that of c -axis of β -iPP are 3.13%, 6.48% and 0.15% respectively, which suggest that

Table III. The cell parameters of β -iPP¹⁶ and the bisamide compounds

	system	a (nm)	b (nm)	c (nm)	α (°)	β (°)	γ (°)
β -iPP	trigonal	1.103	1.103	0.649	90.00	90.00	60.00
DPM	Triclinic	1.472	0.896	0.543	63.56	104.17	103.63
DPS	monoclinic	2.590	0.670	1.530	90.00	130.10	90.00
DPG	triclinic	0.911	0.694	1.121	68.48	124.13	102.99
DPA	triclinic	1.177	0.648	0.826	95.07	90.64	100.68
DPH	Triclinic	1.814	0.971	0.740	121.37	89.05	91.42
DPO	Monoclinic	1.911	0.548	0.916	90.00	96.62	90.00
DPN	Triclinic	1.184	1.108	0.879	33.20	125.34	130.97
DPD	Monoclinic	2.134	1.048	1.834	90.00	99.88	90.00

they have good geometric lattice matching relationships. The lengths of b -axis of other compounds are far from 0.65 nm, and their misfit factors are greater than 15%, for example, the f_m between the length of b -axis of DPO and that of c -axis of β -iPP is 18.43%. It may be the reason that other five compounds cannot act as β -nucleating agents for iPP.

It is widely accepted that the epitaxy occurs between the crystal interfaces of polymer and the nucleating agent. On the other hand, α -form iPP also shows the same 0.65 nm periodicity of c -axis.²⁴ Thus it is not enough for only one-dimensional lattice match between nucleating agent and iPP. The lateral matching should be also checked. It is found that the lengths of a -axis of DPS, c -axis of DPG and a -axis of DPA also have good lattice matching relationship with that of a -axis (or b -axis) of β -iPP. The misfit factors are 14.8%, 1.61% and 6.29%, respectively. For the case of DPS/iPP system, when iPP crystallizes from the molten state, its macromolecular chains can be packed into the periodic cavities of the DPS crystals; and ab -plane of DPS crystals may be in contact with β -form iPP (shown in Figure 4). Similarly, ab -plane of DPA and bc -plane of DPG may work as the contacting planes, respectively.

CONCLUSION

The influence of a family of eight N,N' -diphenyl bisamide compounds on the formation of β -form iPP had been studied by using WAXD, DSC and PLM measurements. Among them, N,N' -diphenyl succinamide (DPS), N,N' -diphenyl glutaramide (DPG) and N,N' -diphenyl adipamide (DPA) can induce the β -crystal structure under quiescent isothermal crystallization. DPS and DPA show higher ability than DPG to induce the formation of β -form while they are at the same concentration level. The hydrogen bond and steric hindrance of carbon chain in this family directly affect their crystalline structures, which definitively influence their β -nucleating effects. The periodic lengths of b -axis of DPS, DPG and DPA all are about 0.65 nm, but those of other compounds are far from this value. Further discussion shows that ab -plane of DPS, bc -plane of DPG and ab -plane of DPA may be in connecting with β -crystal of iPP.

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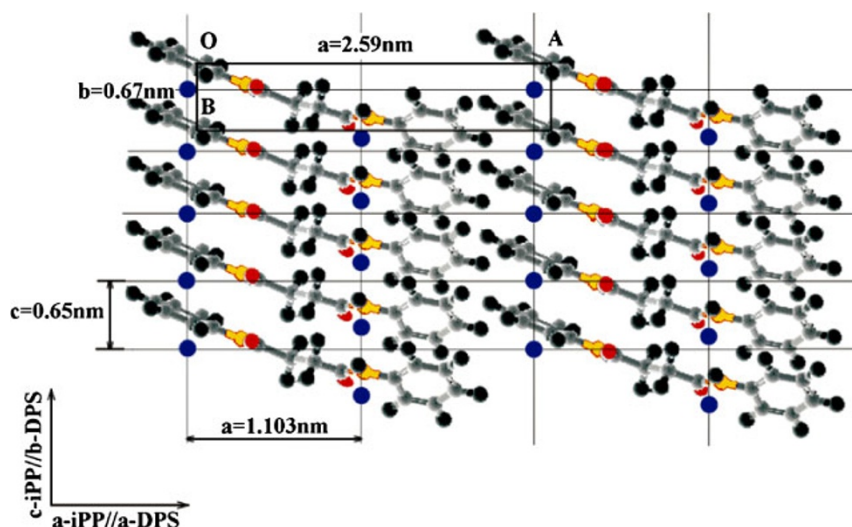


Figure 4. The imaginary contacting plane of β -iPP (ac-plane) and DPS (ab-plane) which was drawn in ball model. ●: CH₃ of iPP; ●: oxygen; ●: nitrogen.

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